

Supplementary Materials: Influence of pH, Temperature and Sample Size on Natural and Enforced Syneresis of Precipitated Silica

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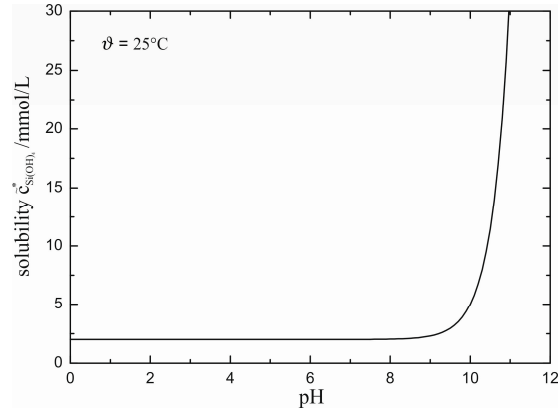


Figure S1. Solubility $\tilde{c}_{\text{Si(OH)}_4}^*$ of monomeric silicic acid as a function of pH, calculated according to Schlomach [1].

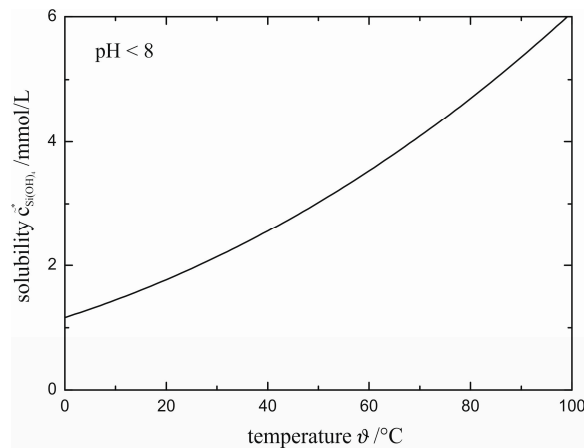


Figure S2. Solubility $\tilde{c}_{\text{Si(OH)}_4}^*$ of monomeric silicic acid as a function of temperature, calculated according to Schlomach [1].

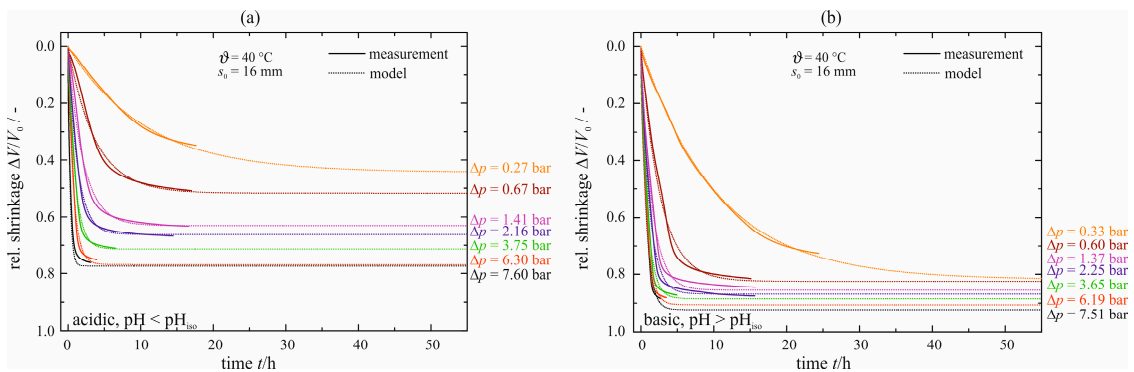


Figure S3. Relative shrinkage $\Delta V/V_0$ of enforced syneresis for $s_0 = 16$ mm and $\vartheta = 40$ °C, acid-catalyzed (a) and base-catalyzed (b).

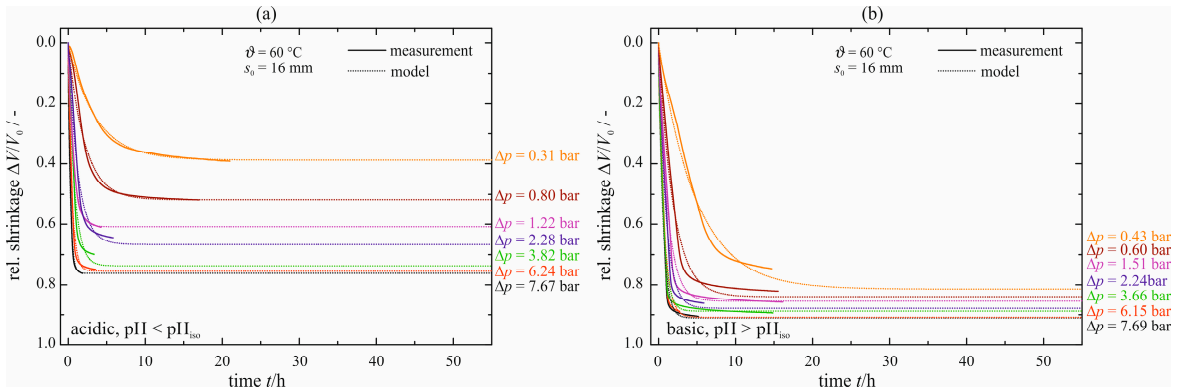


Figure S4. Relative shrinkage $\Delta V/V_0$ of enforced syneresis for $s_0 = 16$ mm and $\vartheta = 60$ °C, acid-catalyzed (a) and base-catalyzed (b).

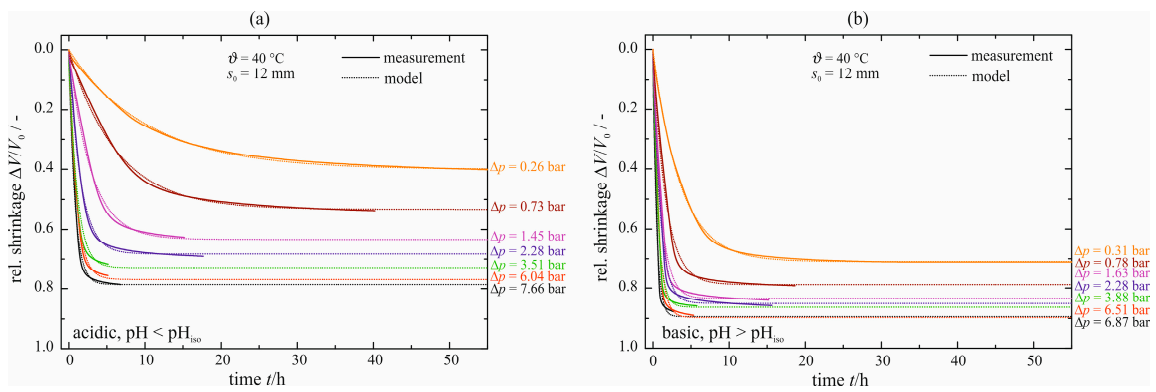


Figure S5. Relative shrinkage $\Delta V/V_0$ of enforced syneresis for $s_0 = 12$ mm and $\vartheta = 40$ °C, acid-catalyzed (a) and base-catalyzed (b).

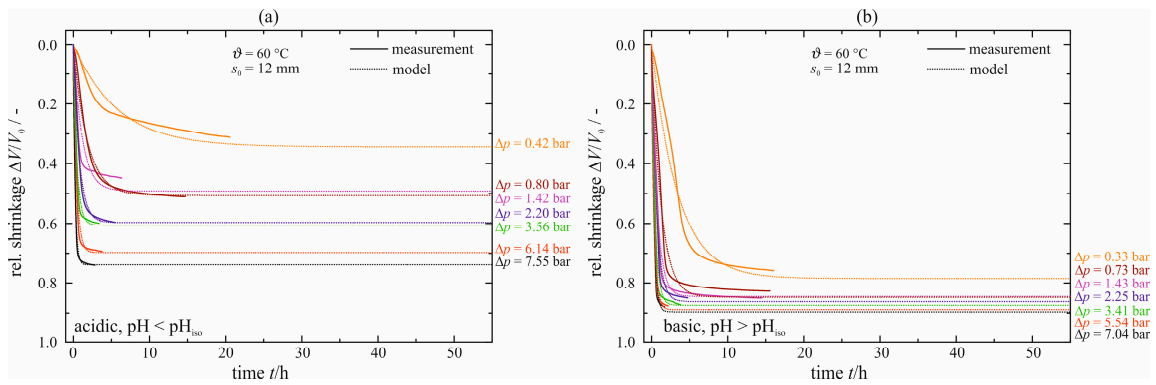


Figure S6. Relative shrinkage $\Delta V/V_0$ of enforced syneresis for $s_0 = 12$ mm and $\vartheta = 60$ °C, acid-catalyzed (a) and base-catalyzed (b).

Table S1. Maximum shrinkage $\Delta V/V_0|_{\max}$ and characteristic time constant τ , enforced syneresis, $s_0 = 16$ mm and $\vartheta = 40$ °C.

Pressure difference $\Delta p/\text{bar}$	Maximum shrinkage $\Delta V/V_0 _{\max}/-$		Time constant τ/h	
	Acid-catalyzed	Base-catalyzed	Acid-catalyzed	Base-catalyzed
	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$
0.27–0.33	0.44	0.82	10.6	10.5
0.60–0.67	0.52	0.82	3.7	2.6
1.37–1.41	0.63	0.85	2.1	1.5
2.16–2.25	0.66	0.87	1.3	1.1
3.65–3.75	0.71	0.88	0.9	0.9
6.19–6.30	0.77	0.91	0.7	0.8
7.51–7.60	0.78	0.92	0.6	0.7

Table S2. Maximum shrinkage $\Delta V/V_0|_{\max}$ and characteristic time constant τ , enforced syneresis, $s_0 = 16$ mm and $\vartheta = 60$ °C.

Pressure difference $\Delta p/\text{bar}$	Maximum shrinkage $\Delta V/V_0 _{\max}/-$		Time constant τ/h	
	Acid-catalyzed	Base-catalyzed	Acid-catalyzed	Base-catalyzed
	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$
0.31–0.43	0.39	0.81	3.5	4.8
0.60–0.80	0.52	0.84	2.1	1.9
1.22–1.51	0.61	0.86	1.7	1.1
2.24–2.28	0.67	0.88	1.2	0.9
3.66–3.82	0.73	0.89	0.8	0.7
6.15–6.24	0.75	0.90	0.5	0.6
7.67–7.69	0.76	0.91	0.3	0.6

Table S3. Maximum shrinkage $\Delta V/V_0|_{\max}$ and characteristic time constant τ , enforced syneresis, $s_0 = 12$ mm and $\vartheta = 20$ °C.

Pressure difference $\Delta p/\text{bar}$	Maximum shrinkage $\Delta V/V_0 _{\max}/-$		Time constant τ/h	
	Acid-catalyzed	Base-catalyzed	Acid-catalyzed	Base-catalyzed
	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$
0.14–0.26	0.40	0.71	10.4	3.8
0.55–0.74	0.53	0.75	6.2	2.2
1.36–1.40	0.63	0.80	2.9	1.3
2.19–2.21	0.68	0.82	1.5	1.1
3.75–3.81	0.74	0.84	1.1	0.8
5.93–6.12	0.77	0.85	1.0	0.6
7.09–7.20	0.79	0.86	0.8	0.5

Table S4. Maximum shrinkage $\Delta V/V_0|_{\max}$ and characteristic time constant τ , enforced syneresis, $s_0 = 12$ mm and $\vartheta = 40$ °C.

Pressure difference $\Delta p/\text{bar}$	Maximum shrinkage $\Delta V/V_0 _{\max}/-$		Time constant τ/h	
	Acid-catalyzed	Base-catalyzed	Acid-catalyzed	Base-catalyzed
	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$
0.26–0.31	0.42	0.71	7.0	3.6
0.73–0.78	0.51	0.78	3.1	1.7
1.45–1.63	0.63	0.83	1.6	1.1
2.28–2.28	0.67	0.85	0.9	0.9
3.51–3.88	0.73	0.86	1.0	0.7
6.04–6.51	0.77	0.89	0.7	0.7
6.87–7.66	0.78	0.89	0.5	0.5

Table S5. Maximum shrinkage $\Delta V/V_0|_{\max}$ and characteristic time constant τ , enforced syneresis, $s_0 = 12$ mm and $\vartheta = 60$ °C.

Pressure difference $\Delta p/\text{bar}$	Maximum shrinkage $\Delta V/V_0 _{\max}/-$		Time constant τ/h	
	Acid-catalyzed	Base-catalyzed	Acid-catalyzed	Base-catalyzed
	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$	$\text{pH} < \text{pH}_{\text{iso}}$	$\text{pH} > \text{pH}_{\text{iso}}$
0.33–0.42	0.35	0.78	5.8	3.4
0.73–0.80	0.51	0.85	1.8	1.2
1.42–1.43	0.50	0.84	1.2	0.7
2.20–2.25	0.60	0.86	0.8	0.6
3.41–3.56	0.61	0.87	0.5	0.4
5.54–6.14	0.70	0.89	0.4	0.4
7.04–7.55	0.74	0.90	0.2	0.3

Table S6. Fitting parameters A – F of Equations (4) and (5) for the acid-catalyzed gel.

Temperature $\vartheta/^\circ\text{C}$	$A/-$	B/bar	C/bar	D/h	$E/\text{h}\cdot\text{bar}$	F/bar
20	0.808	−0.322	0.634	0.642	3.026	0.161
40	0.786	−0.277	0.460	−0.057	3.014	0.175
60	0.749	−0.411	0.749	−0.141	2.127	0.101

Table S7. Fitting parameters A – F of Equations (4) and (5) for the base-catalyzed gel.

Temperature $\vartheta/^\circ\text{C}$	$A/-$	B/bar	C/bar	D/h	$E/\text{h}\cdot\text{bar}$	F/bar
20	0.867	−0.113	0.399	0.469	1.225	0.094
40	0.902	−0.121	0.311	0.445	0.981	0.026
60	0.911	−0.145	0.939	−0.024	1.432	0.108



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